

**Liquidus Temperature and Optical Properties Measurement
by Containerless Techniques**

by

Collin D. Anderson

Intersonics, Incorporated
3453 Commercial Avenue
North Brook, IL 60062

Abstract

Reactive alloy liquidus temperatures measured by conventional, contained techniques are often in error due to reactions with containers and gaseous impurities. This paper describes a new liquidus temperature measurement technique that avoids these problems by employing containerless processing. This technique relies on precise and accurate noncontact temperature measurements (NCTM), which are made possible by spectral emissivity values. The spectral emissivities, ϵ_λ , and are measured along with the optical properties (real, n , and imaginary, k , components of the index of refraction) using polarimetric techniques on electromagnetically levitated specimens. Results from work done at Vanderbilt University and Intersonics on the Ti-Al system are presented to demonstrate the above techniques.

Introduction

There are three introductory slides. The first slide acknowledges NASA funding sources and the presenter's colleagues at Vanderbilt University, Intersonics and Allied-Signal Corporation. The second slide presents an outline of the talk. The third slide shows how true temperatures are determined from measured apparent temperatures and spectral emissivities.

Emissivity and Optical Properties

EXPERIMENTAL: There are two slides in this category. The first slide describes the experimental approach for measuring n , k , and ϵ_λ . The second slide shows two schematic views of the electromagnetic levitation apparatus at Intersonics, where these measurements were made. This slide shows the optical paths of the laser used for polarimetry and the emitted light collected by the pyrometer.

RESULTS: There are three slides showing results for the Ti-Al system. The first two slides show n , k , and $\epsilon_{633\text{ nm}}$ as a function of temperature for three different Ti-Al compositions. The liquidus temperature, T_m , is marked for each composition and delineates

between results for undercooled and superheated melts. The property values are relatively insensitive to temperature in the undercooled regimes but show some temperature dependence in the overheated regimes, particularly for two of the compositions. The third slide shows the composition dependence of n , k , and $\epsilon_{633\text{ nm}}$ in the Ti-Al system. It is clear from the data that NCTM measurements based on simple assumptions about the emissivity dependence on composition would result in substantial errors (at these temperatures, an error of 0.01 in emissivity results in a true temperature error of about 4 K).

Liquidus Temperatures

EXPERIMENTAL: There are two slides in this category. The first slide presents the experimental approach for making containerless liquidus temperature measurements. The second slide is a schematic diagram of the electromagnetic levitation apparatus at Vanderbilt University, where the liquidus temperature measurements on Ti-Al were conducted.

RESULTS: The Ti-Al liquidus results are presented in three slides. The first slide shows a typical overall melting and recalescence cycle for a specimen that was allowed to deeply undercool and recalesce spontaneously. The second slide shows typical temperature-versus-time data for a specimen that had nucleation induced at some small bulk undercooling to determine the liquidus temperature. The third slide is a table summarizing the liquidus temperature measurement results for eight different compositions in the near-equiatomic region of the Ti-Al system. The Table also shows the apparent liquidus temperatures and spectral emissivities that were used to calculate the true liquidus temperatures.

DISCUSSION: There are three slides used to discuss the liquidus results. The first slide is a plot of the present results and three sets of older results from the literature. The second slide presents three more recent sets of results from the literature and compares them to the present results. All of the literature results were determined using contained techniques. The third slide is a phase diagram that was drawn by combining the present liquidus results with solid state data from the literature. The diagram is reasonable in form.

Conclusions

This slide gives two conclusions derived from the present work.

Advances in Technique

The final slide discusses some advances that have been made in the techniques since the completion of the Ti-Al work.

References

1. C.D. Anderson, W.H. Hofmeister, and R.J. Bayuzick: Metall. Trans. A, 1993, Vol. 24A, pp. 61-66.
2. S. Krishnan, C.D. Anderson, J.K.R. Weber, P.C. Nordine, W.H. Hofmeister, and R.J. Bayuzick: Metall. Trans. A, 1992, Vol. 24A, pp. 67-72.
3. H.J. Kostkowski and R.D. Lee: Proc. 4th Symposium on Temperature, It's Measurements and Control in Science and Industry, Columbus, OH March 27-31, 1961, Reinhold Publishing Co., NY, p.1.
4. H.R. Ogden, D.J. Maykuth, W.L. Finlay, and R.I. Jaffe: Trans. AIME, 1951, Vol. 191, pp. 1150-1155.
5. E.S. Bumps, H.D. Kessler, and M. Hansen: Trans. AIME, Vol. 194, 1952, pp. 609-614.
6. I.I Kornilov, E.N. Pylaeva, and M.A. Volkova: Izv. Akad. Nauk SSSR, Otd. Khim. Nauk, Vol. 7, 1956, pp. 771-780 (in Russian).
7. J.C. Schuster and H. Ipser: Z. Metallkd., 1990, Vol. 81, p. 389.
8. J.C. Mishurda and J.H. Perepezko: in Microstructure/Property Relationships in Titanium Aluminides and Alloys, Young-Won Kim and Rodney R. Boyer, eds. TMS Warrendale, PA, 1991, pp. 3-30.
9. S.C. Huang and P.A. Siemers: Metall Trans. A, 1989, Vol. 20A, pp. 1899-1906.
10. C. McCullough, J.J. Valencia, C.G. Levi, and R. Mehrabian, Acta Metall. 37, 1989, 1321.

ACKNOWLEDGEMENTS

THIS APPROACH HAS BEEN DEMONSTRATED ON THE TI-AL SYSTEM AT VANDERBILT UNIVERSITY AND INTERSONICS, INCORPORATED. THIS WORK IS IN PRESS IN METALLURGICAL TRANSACTIONS. A RELATED PAPER WAS RECENTLY PUBLISHED IN METALLURGICAL TRANSACTIONS.

THE AUTHORS OF THE TI-AL WORK ARE:

LIQUIDUS AND OPTICAL PROPERTIES WORK

C.D. ANDERSON (FORMERLY OF VANDERBILT), W.H. HOFMEISTER AND R.J. BAYUZICK OF VANDERBILT

OPTICAL PROPERTIES WORK

S. KRISHNAN, J.K.R. WEBER, AND P.C. NORDINE OF INTERSONICS, INCORPORATED AND ABOVE THREE AUTHORS

FUNDING FOR THE TI-AL WORK WAS PROVIDED BY THE NASA OFFICE OF COMMERCIAL PROGRAMS UNDER GRANT NAGW-810 AND THE NASA MICROGRAVITY SCIENCES AND APPLICATIONS DIVISION, GRANTS NAG8-765 AND NAS8-37472. SOME ALLOYS AND ANALYTICAL WORK WERE PROVIDED BY DAVID SKINNER OF ALLIED-SIGNAL CORPORATION.

OUTLINE

1. NON-CONTACT TEMPERATURE MEASUREMENT REVIEW
2. OPTICAL PROPERTIES
 - BACKGROUND AND APPARATUS
 - RESULTS AND DISCUSSION
3. LIQUIDUS TEMPERATURES
 - TECHNIQUE
 - RESULTS AND DISCUSSION
4. CONCLUSIONS
5. ADVANCES IN TECHNIQUE

NON-CONTACT TEMPERATURE MEASUREMENT (NCTM)

$$\frac{1}{T_t} = \frac{1}{T_{app}} + \frac{\lambda_{eff}}{C_2} \ln \epsilon_\lambda$$

WANT TO DETERMINE: T_t = TRUE TEMPERATURE

KNOWN: C_2 = PLANCK'S SECOND CONSTANT
 λ_{eff} = EFFECTIVE WAVELENGTH OF PYROMETER (FROM CALIBRATION)

MEASURE WITH PYROMETER: T_{app} = APPARENT TEMPERATURE AT λ

IN GENERAL, ϵ IS A FUNCTION OF λ , T, AND COMPOSITION.

ϵ_λ = NORMAL SPECTRAL EMISSIVITY AT λ (IS THE PAST WAS FOUND BY CALIBRATION OR BY INTERPOLATION BETWEEN PURE ELEMENT VALUES.)

IN THE PRESENT STUDY, ϵ_λ IS MEASURED WITH ROTATING ANALYZER ELLIPSOMETER AT 633 nm.

EMISSIVITY MEASUREMENT APPROACH

1. LEVITATE, MELT, AND OVERHEAT SAMPLE.
2. AT TEMPERATURE OF INTEREST, SHINE A PLANE-POLARIZED LASER (USED He-Ne, λ = 632.8 nm) FROM SAMPLE SURFACE AND MEASURE CHANGE IN POLARIZATION OF LASER DUE TO REFLECTION.
3. FROM THIS CHANGE, DETERMINE INDICES OF REFRACTION, n and k, FOR SAMPLE.
4. CALCULATE NORMAL SPECTRAL EMISSIVITY FROM n AND k.

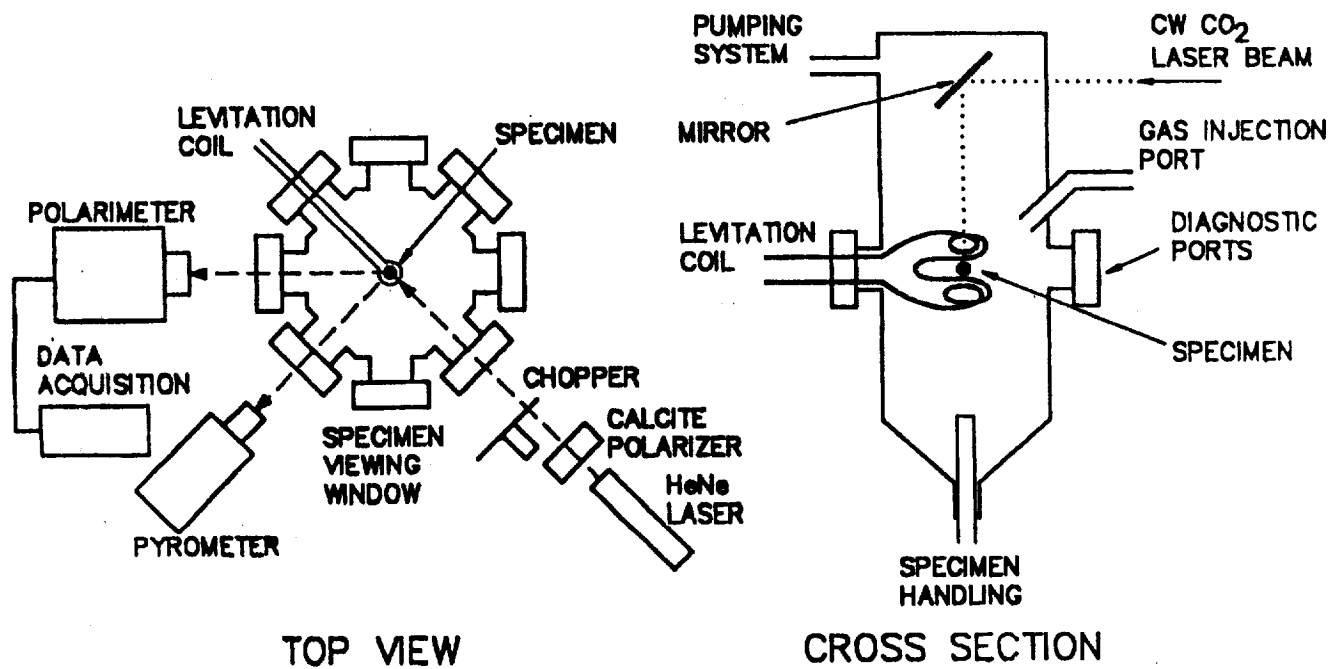
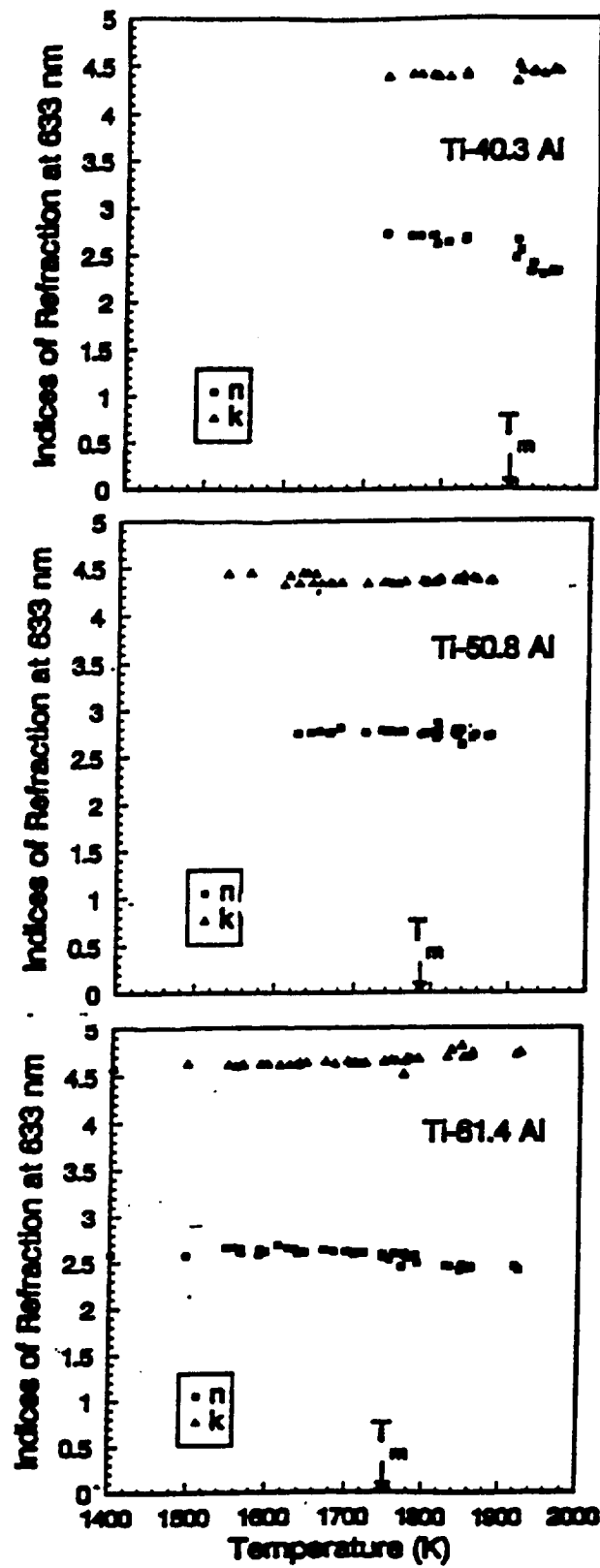
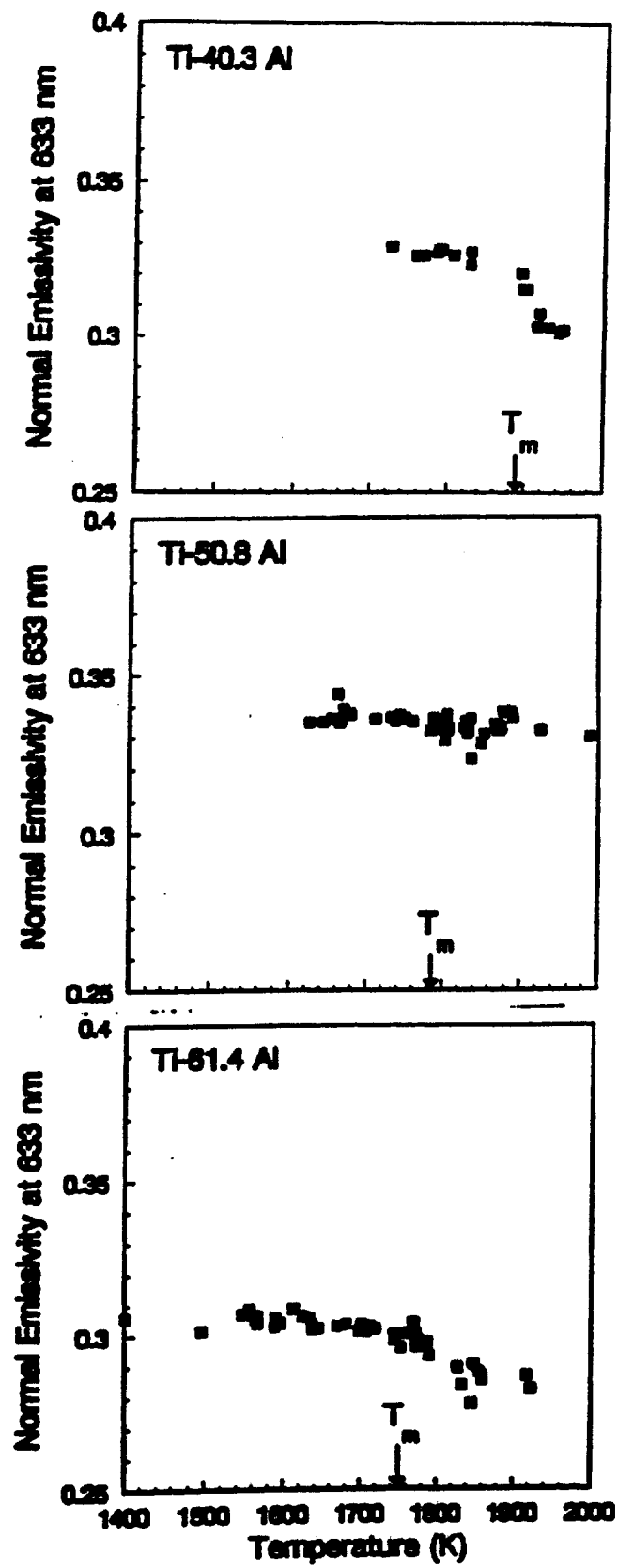


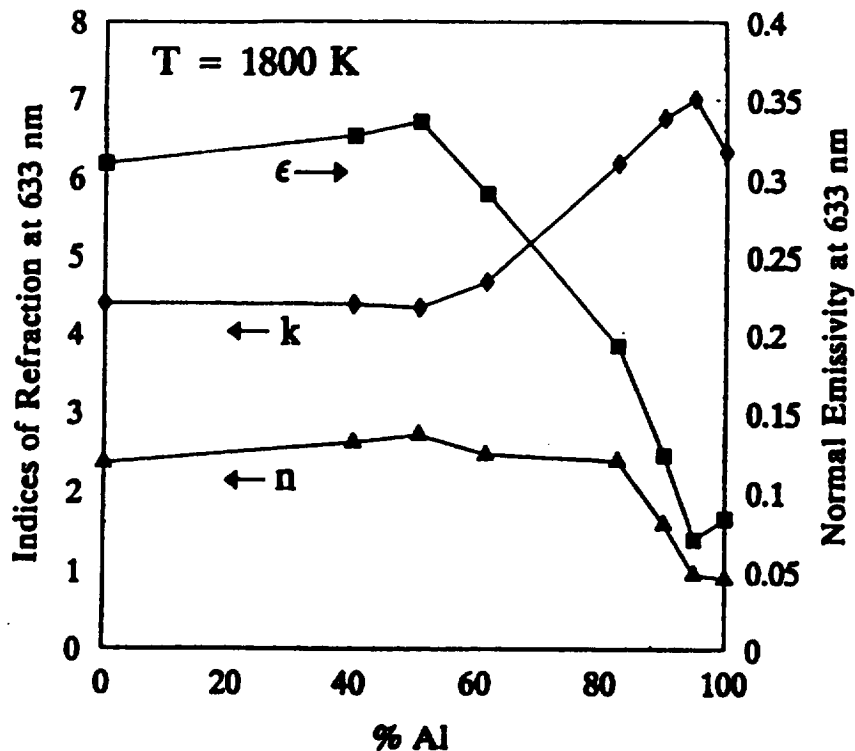
Figure 2 - Schematic diagram of electromagnetic levitation apparatus and laser polarimeter at Intersonics, Incorporated.



Indices of Refraction as a Function of Temperature for 3 Ti-Al Compositions.



Normal Spectral Emissivities at 633 nm as a Function of Temperature for 3 Ti-Al Compositions.



Emissivities and Indices of Refraction vs. Composition

LIQUIDUS TEMPERATURE DETERMINATION APPROACH

1. LEVITATE, MELT, AND OVERHEAT TO CLEAN SAMPLE
2. COOL TO VICINITY OF ESTIMATED T_{liq} .
3. TOUCH SAMPLE WITH AMORPHOUS WHISKER.
4. IF SAMPLE DOES NOT SOLIDIFY (i.e. NO RECALESCENCE), $T_{\text{failure}} > T_{\text{liq}}$.
5. COOL SAMPLE TO SLIGHTLY LOWER TEMPERATURE AND REPEAT UNTIL SOLIDIFICATION OCCURS AT SOME SMALL UNDERCOOLING (INDICATED BY SMALL RECALESCENCE PEAK).
 $T_{\text{rise}} < T_{\text{liq}}$

LOWEST T_{failure} PROVIDES UPPER BOUND.

HIGHEST T_{rise} PROVIDES LOWER BOUND.

ACCURACY AND PRECISION DETERMINED BY NCTM.

BY BRACKETING A SMALL NUMBER OF SAMPLES, IT WAS FOUND THAT T_{rise} (PEAK TEMPERATURE AFTER RECALESCENCE) FOR SMALL BULK UNDERCOOLINGS $\sim T_{\text{liq}}$.

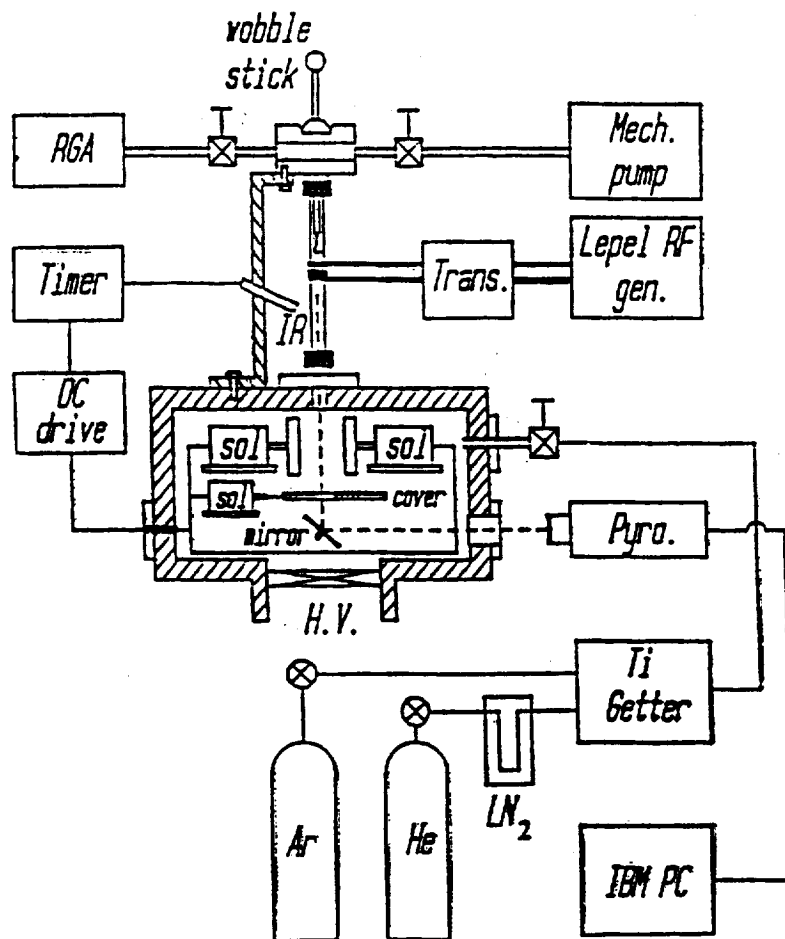
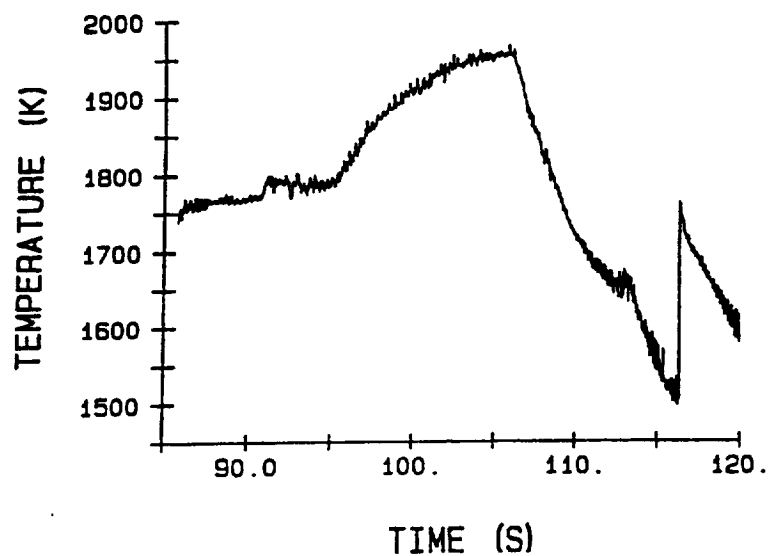
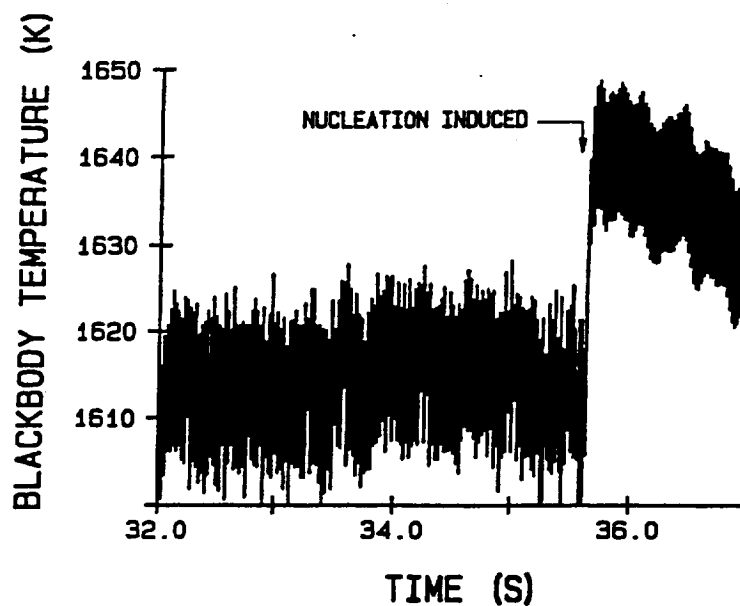


Figure 1 – Schematic diagram of electromagnetic levitation apparatus at Vanderbilt University.



Typical Overall Melting and Recalescence Cycle.

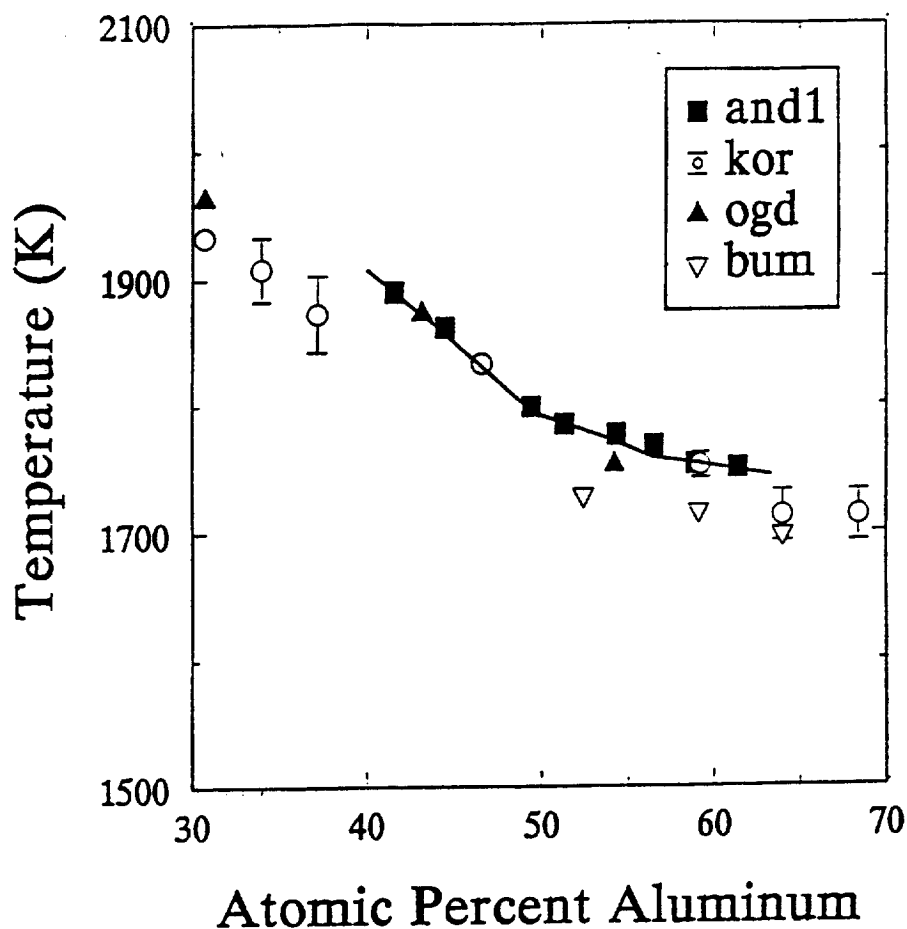


Typical Temperature-vs-Time Data Showing Recalescence Resulting From Induced Nucleation of a Slightly Undercooled Ti-49.6 at.% Al Alloy.

TABLE 1

LIQUIDUS TEMPERATURES FOR TITANIUM-ALUMINUM ALLOYS

ATOMIC % Al ± 1.0	T _{Liq} (K) APPARENT	EMISSIONITY ± .015	T _{Liq} (K) TRUE
41.6	1717 ± 6	0.320	1890 ± 10
44.5	1696 ± 6	0.324	1862 ± 10
49.4	1647 ± 8	0.332	1799 ± 11
51.3	1635 ± 8	0.333	1785 ± 11
54.3	1625 ± 8	0.324	1777 ± 11
56.5	1615 ± 8	0.318	1768 ± 11
58.9	1601 ± 8	0.311	1754 ± 11
61.4	1594 ± 10	0.301	1751 ± 14



Comparison of Current Liquidus Data with Literature

RECENT NEAR-EQUIATOMIC TIAI LIQUIDUS RESULTS

1. **SCHUSTER AND IPSER (BY DTA)**

0 TO 20 K LOWER THAN PRESENT RESULTS.

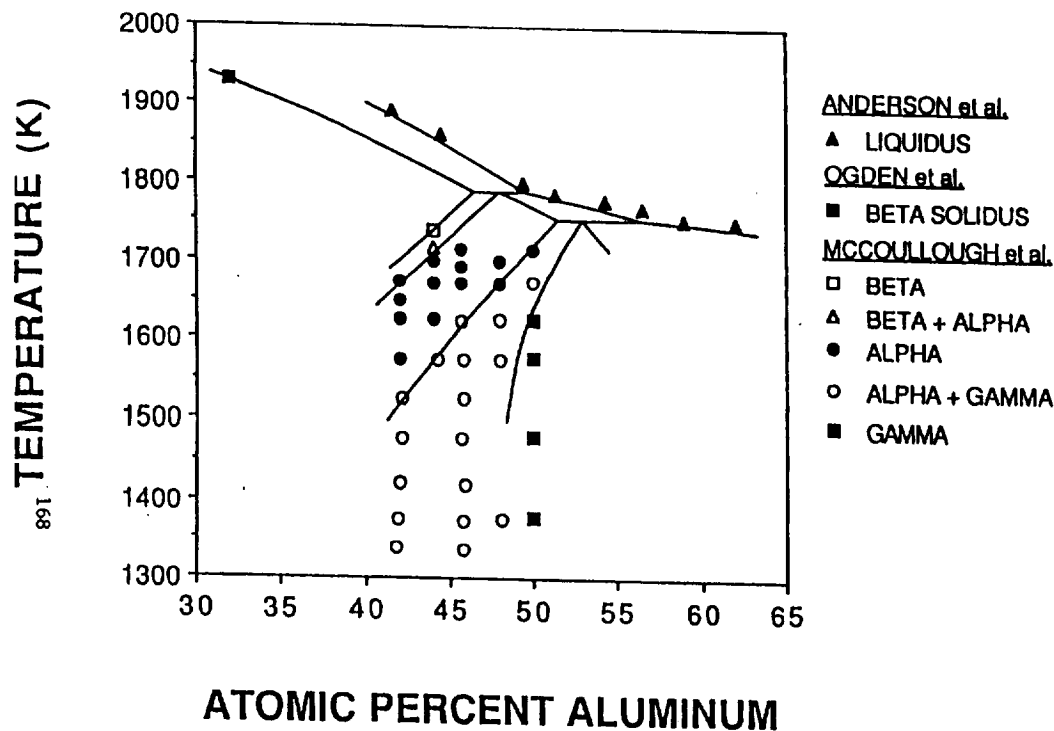
2. **MISHURDA AND PEREPEZKO (BY DTA)**

20 TO 35 K LOWER THAN PRESENT RESULTS.

3. **HUANG AND SEIMERS (BY OBSERVING MICROSTRUCTURES AFTER FURNACE TREATING)**

40 TO 50 K LOWER THAN PRESENT RESULTS.

CONTAINED TECHNIQUES WERE USED IN ALL OF THESE STUDIES.



Current Liquidus Temperatures Plotted with 1 Set of Literature Solid-State Data by McCullough et al.

CONCLUSIONS AND IMPLICATIONS FROM Tl-Al WORK

1. ASSUMPTIONS ABOUT COMPOSITION DEPENDENCE OF ϵ CAN LEAD TO SERIOUS TEMPERATURE MEASUREMENT ERRORS.
2. DESCRIBED TECHNIQUE PROVIDES ACCURATE LIQUIDUS TEMPERATURES.

ADVANCES IN TECHNIQUE

IN THE PAST:

- EMISSIVITIES AND LIQUIDUS TEMPERATURES MEASURED AT TWO DIFFERENT TIMES USING TWO DIFFERENT APPARATUSES.
- ACCURACY DEPENDS ON SAMPLES HAVING SAME SURFACE CONDITION FOR THE TWO MEASUREMENTS.

WITH USE OF NEW DIVISION-OF-AMPLITUDE POLARIMETER (DOAP) AND WITH CHANGE IN EM CHAMBER DESIGN AT INTERSONICS:

- CAN MAKE BOTH MEASUREMENTS SIMULTANEOUSLY.
- IN THIS WAY, MEASUREMENT IS INDEPENDENT OF SAMPLE SURFACE STATE.

